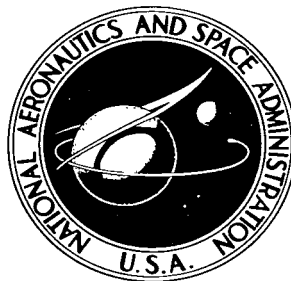


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FRICITION AND WEAR OF NICKEL-ALUMINUM ALLOYS AND SOME SULFUR-MODIFIED STEELS IN VACUUM TO 10^{-9} MILLIMETER OF MERCURY

by Donald H. Buckley and Robert L. Johnson

Lewis Research Center

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SUMMARY

The friction, wear, and welding characteristics of 52100, 440-C stainless steel, and M-2 tool steel with and without the addition of 0.4 to 0.5 percent sulfur were studied in vacuum (10^{-9} mm Hg). Studies were also conducted with simple nickel-aluminum binary alloys in vacuum.

Friction and wear studies were made with a hemispherical (3/16-in.-rad.) rider, which slides in a circular path on the flat surface of a rotating metal disk of the same metal. The specimens in vacuum had a load of 1000 grams (2.2 lb), a sliding velocity of 75 to 1960 feet per minute, and a temperature of 75° F.

The addition of 0.4 to 0.5 percent sulfur to 52100, 440-C, and M-2 reduced friction, wear, and welding normally encountered with these alloys in vacuum. With nickel-aluminum binary alloys friction and wear improved with the addition of aluminum to nickel. A 16.4-percent-aluminum - nickel alloy exhibited lower friction and less wear and metal transfer in vacuum than did two commercial nickel-base alloys.

INTRODUCTION

Space-vehicle components requiring lubrication should be made of materials having inherently good friction and wear characteristics. Many of the failures experienced with such components as gears, bearings, and seals might not have occurred if the materials had possessed some self-lubricating properties. Under conditions of inadequate lubrication, where metal-metal contact occurs, such materials would not exhibit the high friction, wear, metal transfer, welding, and seizure encountered with conventional alloys.

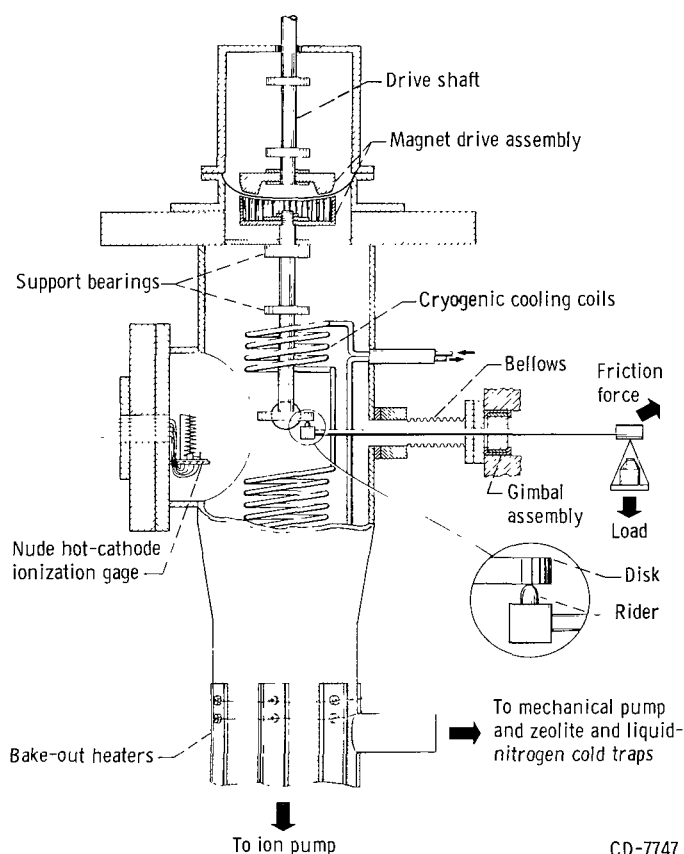
One approach to reducing the friction and wear properties of lubricated materials is to use powder metallurgical techniques to produce bodies with a solid film lubricant incorporated within the structure (refs. 1 to 4). Another

approach is the use of porous bodies impregnated with lubricating materials (e.g., cotton-cloth-laminated phenolics with oils, ref. 5). In each of these approaches the lubricating phase and the structural phase retain their individual identity. These materials, however, generally suffer from poor mechanical strength, which limits their application.

Another approach to obtaining self-lubricating materials for structural components of lubricated systems is that of incorporating the lubricating constituents in the alloy (e.g., lead as the lubricant in lead-tin-bronze bearing materials). Such structures have much better mechanical strength than either pressed or porous bodies. This approach was taken in the investigation of reference 5 with simple binary alloys in an attempt to establish the lubricating characteristics of various structures and materials alloyed with pure metals. The results of the investigation indicated that the addition of materials such as sulfur to iron and oxygen and tin to nickel appreciably reduced friction, wear, and metal transfer.

The general objective of this investigation was to apply the concepts utilized in that of reference 5 with simple binary alloys to more complex alloys that may have potential in actual structural components of lubricated systems. The specific objectives of the investigation were as follows:

- (1) To prepare alloys of 52100, 440-C stainless steel, and M-2 tool steel modified by the addition of approximately 0.5 percent sulfur
- (2) To prepare simple binary nickel-aluminum alloys with desirable mechanical properties and phases imparting good lubricating characteristics
- (3) To determine the friction and wear characteristics of these alloys in vacuum (10^{-9} mm Hg)



APPARATUS

The apparatus used in this investigation is described in detail in references 6 and 7 and is shown in figure 1. The basic elements of the apparatus were the specimens (a $2\frac{1}{2}$ -in.-diam. flat disk and a $3/16$ -in.-rad. rider) mounted in a vacuum chamber. The

Figure 1. - High-vacuum friction and wear apparatus.

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end of the shaft opposite the magnet contained the disk specimen.

The rider specimen was supported in the chamber by an arm that was mounted by gimbals and bellows to the chamber. A linkage at the extended end of the retaining arm was connected to a strain-gage assembly. The assembly was used to measure frictional force. Load was applied through a dead-weight loading system. The rider specimen was generally loaded against the disk surface with a 1000-gram load, and friction experiments were conducted at sliding velocities of 75 to 1960 feet per minute.

Attached to the lower end of the specimen chamber was a 400-liter-per-second ionization pump and a mechanical forepump with zeolite and liquid-nitrogen cold traps. The pressure in the chamber was measured adjacent to the specimen with a nude hot-cathode ionization gage. In the same plane as the specimens and the ionization gage was a diatron-type mass spectrometer (not shown in fig. 1) for determining the gases present in the vacuum system. A 20-foot 3/16-inch-diameter stainless-steel coil was used for liquid-nitrogen and liquid-helium cryopumping of the vacuum system.

SPECIMEN PREPARATION

Casting Procedure

The alloys used in this investigation included 52100, 440-C stainless steel, and M-2 tool steel with 0.4 to 0.5 percent sulfur added and a series of nickel-aluminum binary alloys. All specimen preparation was done at the Lewis Research Center.

The 52100, 440-C stainless steel, and M-2 tool steel were prepared by the addition of iron sulfide slugs to the alloys. The iron sulfide slugs were packed in the center of a zirconium oxide crucible, and the respective alloys were packed about the iron sulfide. The furnace was then evacuated to 10^{-4} millimeter of mercury. The chamber was filled with dry argon, and the temperature was raised to melt the charge. After the material melted, it was poured into a cold copper mold and was allowed to cool to room temperature.

The nickel-aluminum binary alloys were prepared by the addition of pure aluminum to electrolytic nickel. The melting and casting procedure used was the same as that employed with the iron-base sulfur alloys.

The castings from the melts were machined into specimens, and samples were taken for chemical analysis and metallographic examination. Chemical analyses for the concentrations of sulfur and aluminum were made. The concentrations of sulfur in the iron alloys were as follows:

Alloy	Sulfur added, percent	Sulfur in alloy, percent	Hardness, R _C
440-C	0	0.02	56
Sulfur-modified 440-C	.5	.44	57.5
52100	0	.025	60
Sulfur-modified 52100	.5	.41	60
M-2	0	-----	65
Sulfur-modified M-2	.5	.5	65

The concentrations of aluminum in nickel were as follows:

Alloy	Aluminum added, percent	Aluminum in alloy, percent	Hardness, R _C
5.5-Percent-aluminum-nickel	6.0	5.53	^a Av., 10.4 Ni ₃ Al, 24
13.2-Percent-aluminum-nickel	14.0	13.24	^a Av., 15.3 Ni ₃ Al, 24
16.4-Percent-aluminum-nickel	16.6	16.40	^a Av., 36 Ni ₃ Al, 24
27.1-Percent-aluminum-nickel	28.0	27.10	40

^aR_C equivalent.

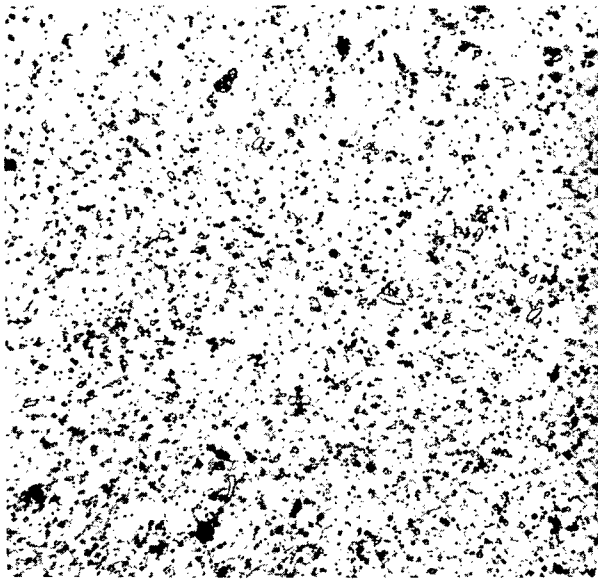
Specimen Finish and Cleaning Procedure

For friction and wear experiments the disk and rider specimens were of the same material and were finish ground to 4 to 8 microinches. Before each experiment the disk and the rider were given the same preparatory treatment: (1) a thorough rinsing with acetone to remove oil and grease, (2) a polishing with moist levigated alumina on a soft polishing cloth, and (3) a thorough rinsing with tap water followed by distilled water. For each experiment a new set of specimens was used.

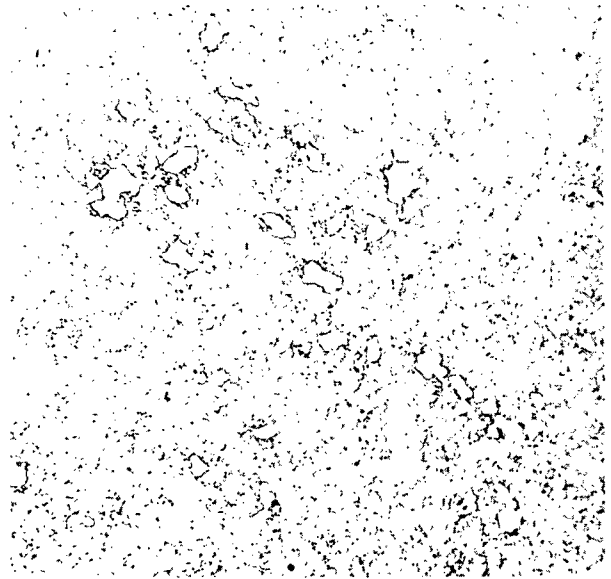
RESULTS AND DISCUSSION

Sulfur in Ferrous Alloys

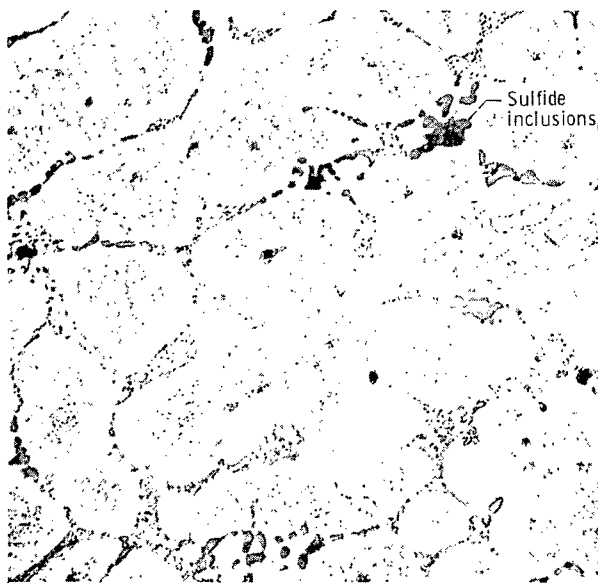
Because improved friction, wear, and nonwelding tendencies were obtained with binary iron-sulfur alloys in the investigation of reference 6, some bear-



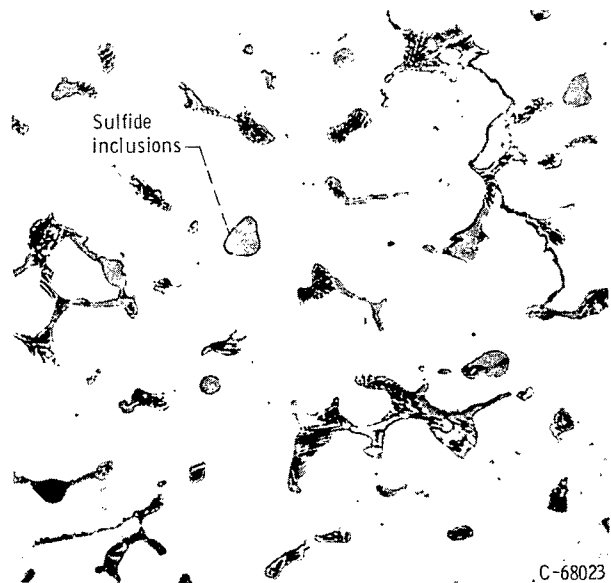
(a) M-2 (wrought).



(b) 440-C (wrought).



(c) Sulfur-modified M-2 (cast).



(d) Sulfur-modified 440-C (cast).

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Figure 2. - Photomicrographs of M-2 tool steel and 440-C stainless steel modified by addition of 0.4 to 0.5 percent sulfur. X500.

ing alloys were cast with the addition of sulfur to their structure. Photomicrographs for two of these modified alloys, M-2 tool steel and 440-C stainless steel, are presented in figure 2 with photomicrographs for the standard alloys. The standard wrought alloys have a fine grain structure. In contrast, the alloys cast with the sulfide inclusion have a very coarse grain structure, as shown in figures 2(c) and (d). With the modified M-2 composition the sulfide inclusions appear primarily in the grain-boundary regions of the alloy, while with 440-C stainless steel the sulfides appear to be dispersed throughout the structure as well as at the grain boundaries. After being machined into specimens and just before being finish ground, the sulfur-modified alloys were given the same heat treatment as prescribed for the standard alloys. There was essentially no change in hardness between cast and wrought structures.

The friction and wear characteristics of these alloys were next determined in vacuum (10^{-9} mm Hg). The results obtained are presented in figure 3. The friction and wear experiments were conducted with standard wrought alloys, the same alloys in cast form, and sulfur-modified cast alloys. The standard alloy composition in cast form showed very little change in friction and wear (with the exception of 52100 wear) from those for the standard wrought structures. In general, all three compositions showed an improvement in friction and wear with the addition of sulfur to the structure (fig. 3). The most marked improvement in friction was noted with 52100. Initial values of 0.68 for the wrought structure were obtained with residual oxides present. With wearing

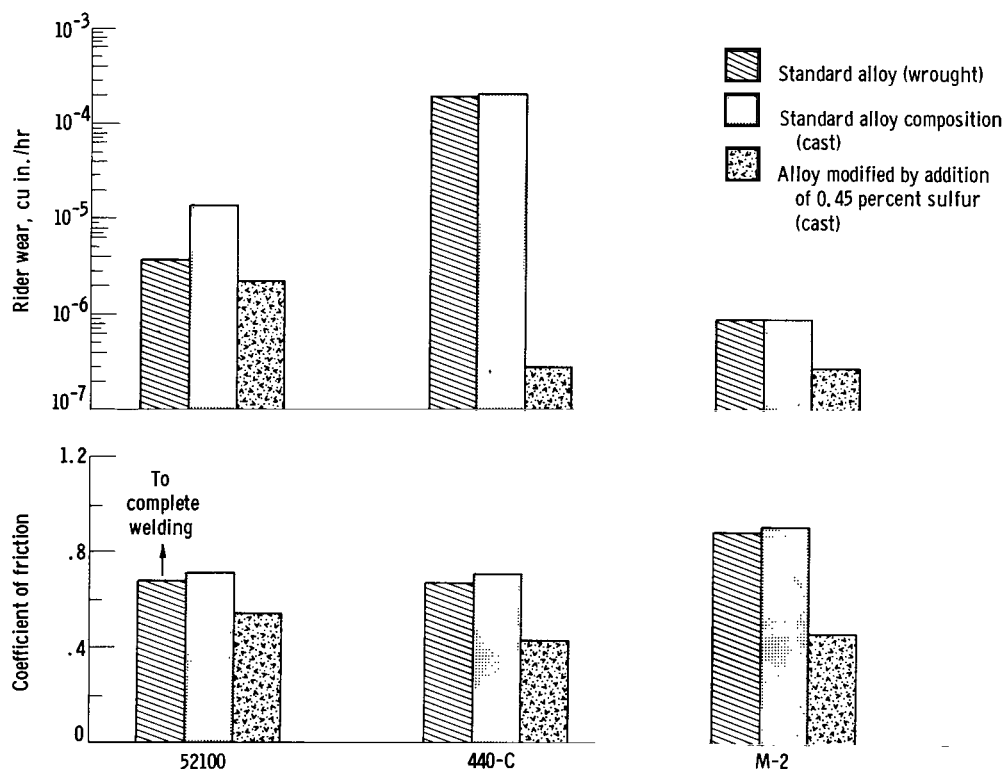


Figure 3. - Coefficient of friction and rider wear for sulfur-modified alloys in vacuum (10^{-9} mm Hg). Sliding velocity, 390 feet per minute; load, 1000 grams; ambient temperature, 75° F; duration, 1 hour; disk and rider of same material.

away of the surface oxides, complete welding of the metal was observed. A friction value of 0.54 was obtained with the sulfur-modified structure. The greatest reduction in wear, however, was observed with 440-C stainless steel, for which a difference in wear of nearly three orders of magnitude resulted with the addition of sulfur to the cast composition. Photomicrographs and surface profile traces of the disk wear areas are presented in figure 4. It is of

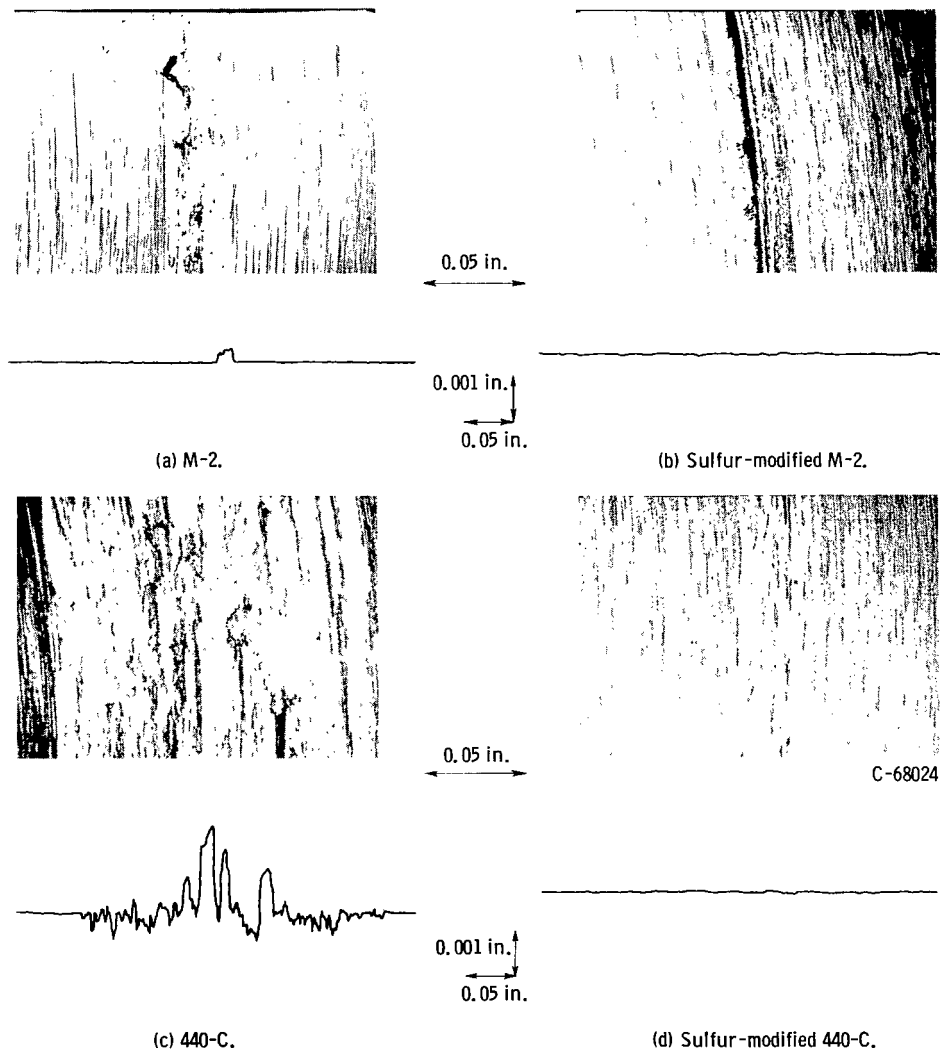


Figure 4. - Photomicrographs and surface profile traces of wear areas in M-2 tool steel and 440-C stainless-steel disks. Sliding velocity, 390 feet per minute; load, 1000 grams; ambient pressure, 10^{-9} millimeter of mercury; duration, 1 hour; disk and rider of same material.

interest to note that metal transfer was observed with the standard wrought M-2 and 440-C alloys. With the addition of sulfur, however, no metal transfer was noted for either of the two alloys. In figure 4 the magnification of the surface profile traces was 1000 times; this was increased to 20,000 times, and still there was no evidence of metal transfer.

The mechanism responsible for the reduction in friction, wear, and metal transfer is the formation of a protective surface film on the alloy by the smearing out of the sulfide phases over it. (Sulfide tests indicated a high concentration of sulfide film in the wear track.) The sulfide surface film substitutes for normal metal oxides in preventing gross metal contact from occurring. As discussed in reference 6, an optimum sulfide concentration for adequate surface protection probably exists.

Since the role of the sulfide in the alloy is to provide a protective surface film, some friction and wear experiments were conducted with only one of the two specimens modified by the addition of sulfur in order to determine whether it was necessary for both to contain sulfur. It was anticipated that an effective film might be provided by one specimen. The results obtained in these experiments together with the data for riders and disks of the same material are presented in figure 5. With a sulfur-modified 440-C rider sliding on

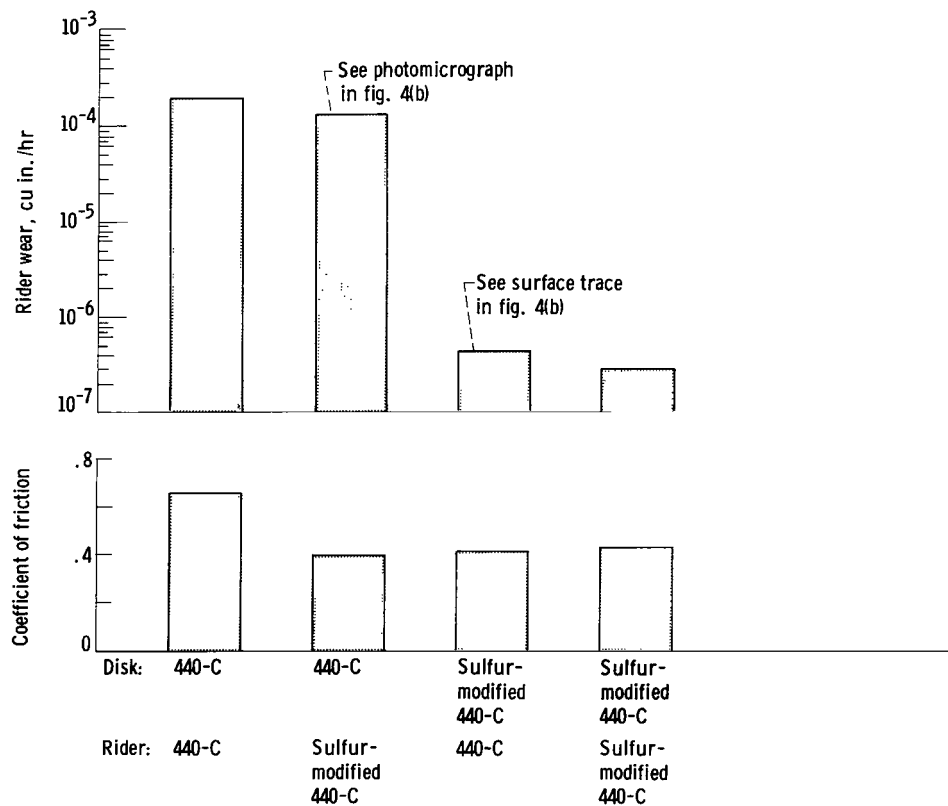


Figure 5. - Coefficient of friction and rider wear for 440-C and sulfur-modified 440-C riders and disks in vacuum (10^{-9} mm Hg). Sliding velocity, 390 feet per minute; load, 1000 grams; ambient temperature, 75° F; duration, 1 hour.

a standard 440-C disk, sufficient sulfide was continuously present at the metal interface to reduce the friction from 0.66 to 0.40; however, the amount of sulfide present was not adequate to reduce the rider wear appreciably. When the specimen combination was reversed (a standard 440-C rider on a sulfur-modified disk), the friction was also about 0.4; the rider wear, however, decreased to a value near that obtained with a sulfur-modified rider on a sulfur-modified

disk. Photomicrographs and surface profile traces for these two specimen combinations are presented in figure 6.

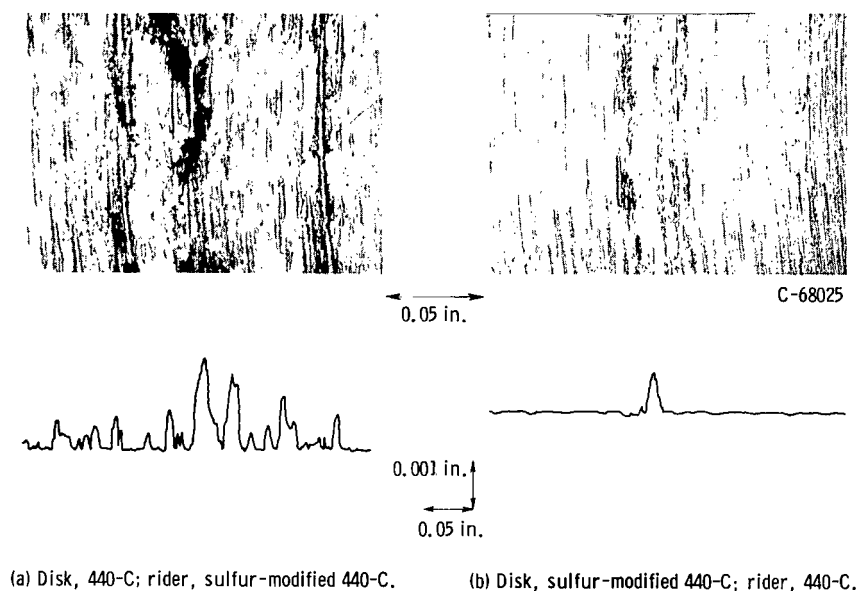


Figure 6. - Photomicrographs and surface profile traces of wear areas in 440-C and sulfur-modified 440-C disks. Sliding velocity, 390 feet per minute; load, 1000 grams; ambient pressure, 10^{-9} millimeter of mercury; duration, 1 hour.

Some experiments at higher sliding velocity were conducted in vacuum with standard and sulfur-modified M-2 to determine the protective effectiveness of the sulfide film. Factors of concern were (1) the ability of the film to be retained and replenished at the interface, (2) the possibility of decomposition or dissociation of the sulfide film in vacuum at higher sliding velocities (higher surface temperatures), and (3) the possibility of a change in the state of the surface film that might influence friction characteristics. The results of these experiments are presented in figure 7. The shape of the curve for the

sulfur-modified M-2 approximates that obtained with the standard M-2 composition, with a general decrease in friction for the modified M-2 composition.

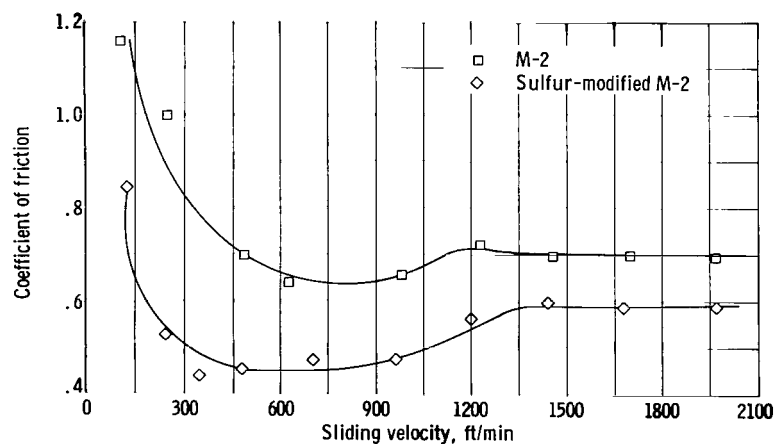


Figure 7. - Coefficient of friction for M-2 and sulfur-modified M-2 in vacuum (10^{-9} mm Hg). Load, 1000 grams; ambient temperature, 75° F; disk and rider of same material.

These results would seem to indicate that no marked changes in the sulfide-surface film occur over the range of sliding velocities investigated in figure 5. Although a general reduction in friction was observed with the sulfur-modified composition,

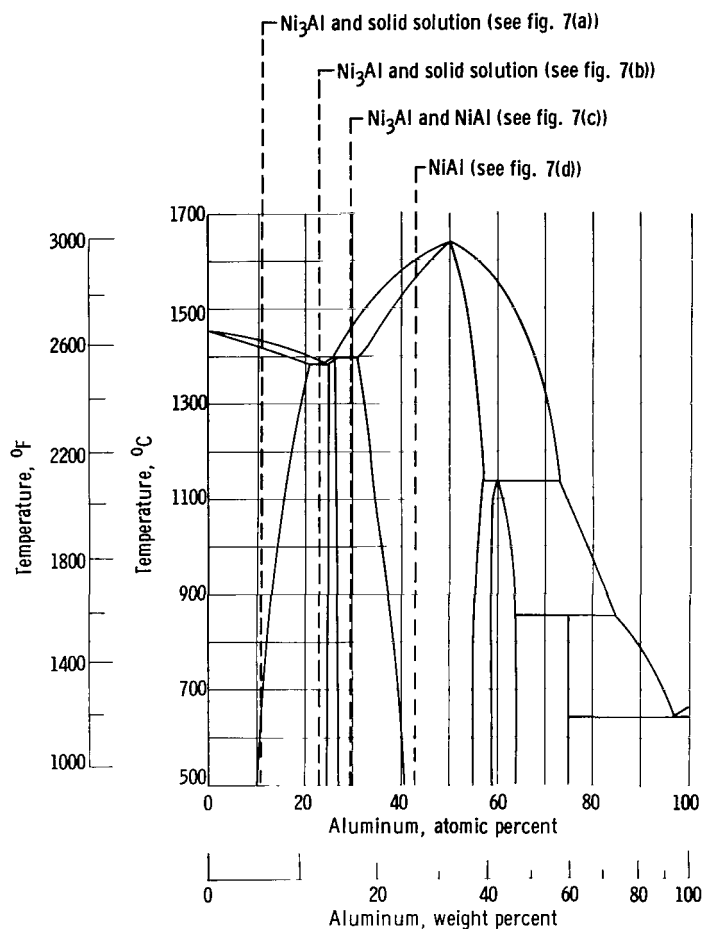


Figure 8. - Nickel-aluminum phase diagram.

compositions were prepared with the intermetallic Ni_3Al in a nickel matrix. An alloy composition of Ni_3Al was prepared. A fourth composition of NiAl was obtained commercially. Photomicrographs of the structures are presented in figure 9. The compositions contained 5.5, 13.3, 15.4, and 27.1 percent aluminum. These compositions gave two alloys with an increasing concentration of intermetallics (duplex structure) and a straight simple intermetallic NiAl .

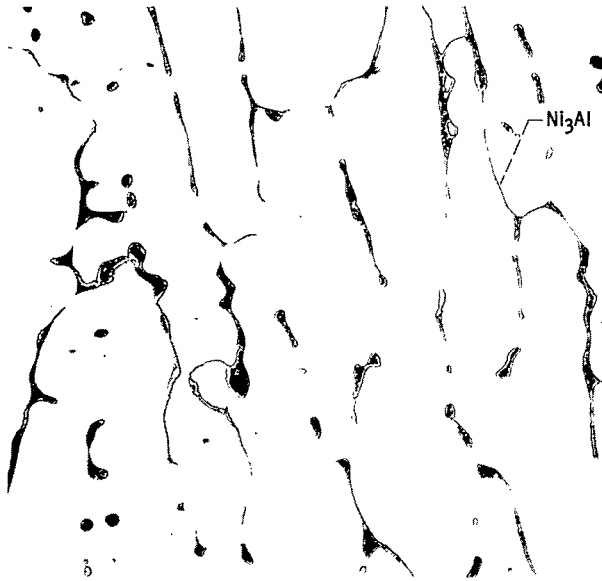
Friction and wear experiments were conducted in vacuum with the four nickel-base alloys. The results obtained in these experiments are presented in figure 10. The friction coefficient did not decrease with the increase in the Ni_3Al compound obtained in going from 5.5 to 13.3 percent aluminum. At 16.4 percent aluminum, however, a decrease in friction was observed.

The rider wear, however, continued to decrease with an increase in percent of the intermetallic present. When, however, sufficient aluminum was added to the alloy so that only the intermetallic compound NiAl was present, the rider wear increased slightly, but a slight reduction in friction was observed. In figure 11 surface profile traces are presented with photomicrographs for the various alloys. Visual observations showed that metal transfer to the disk

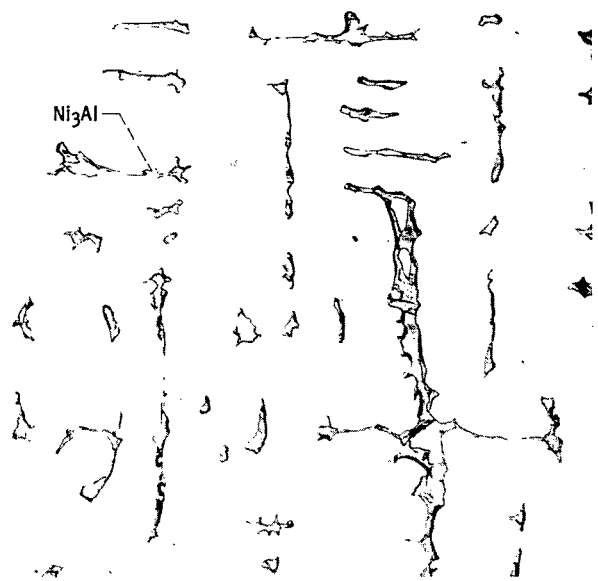
the friction was not low. This result, however, is consistent with observations of iron sulfide at this laboratory and elsewhere. Iron sulfide can, however, reduce appreciably wear and metal transfer, as indicated in figures 3 and 4 (pp. 6 and 7).

Binary Nickel-Aluminum Alloys

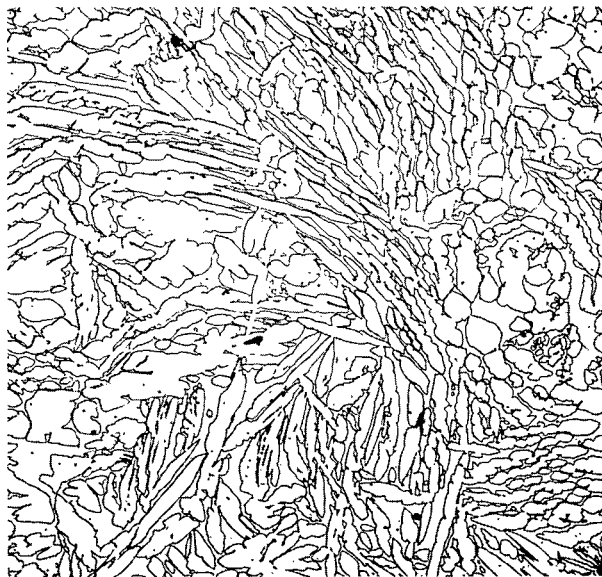
Recently, consideration has been given to nickel-base alloys for some bearing applications (ref. 8). These alloys consist of a number of constituent elements. It was believed that good mechanical properties and improved friction and wear characteristics could be obtained with simple binary nickel alloys. Examination of the phase diagrams for nickel alloys indicated that the binary nickel-aluminum system would be of interest. The phase diagram obtained from reference 9 is presented in figure 8. It is possible to obtain two intermetallic compounds of nickel and aluminum, Ni_3Al and NiAl . Based on the phase diagram, two alloy



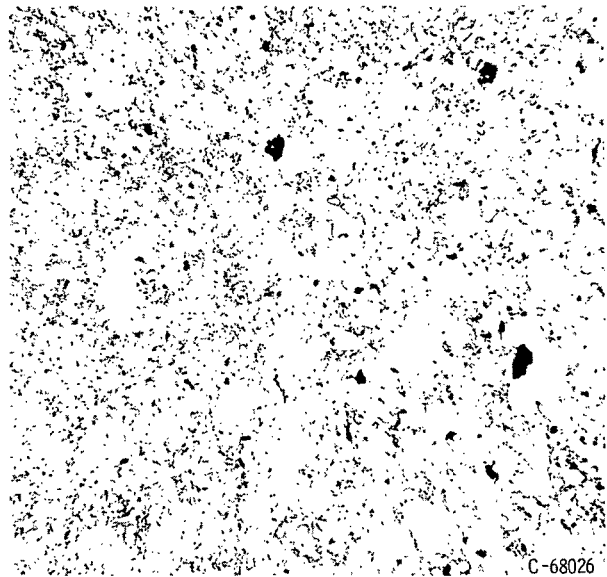
(a) 5.5 Percent aluminum in nickel.



(b) 13.3 Percent aluminum in nickel.



(c) 16.4 Percent aluminum in nickel.



(d) 27.1 Percent aluminum in nickel.

Figure 9. - Photomicrographs of binary nickel-aluminum alloys. X250.

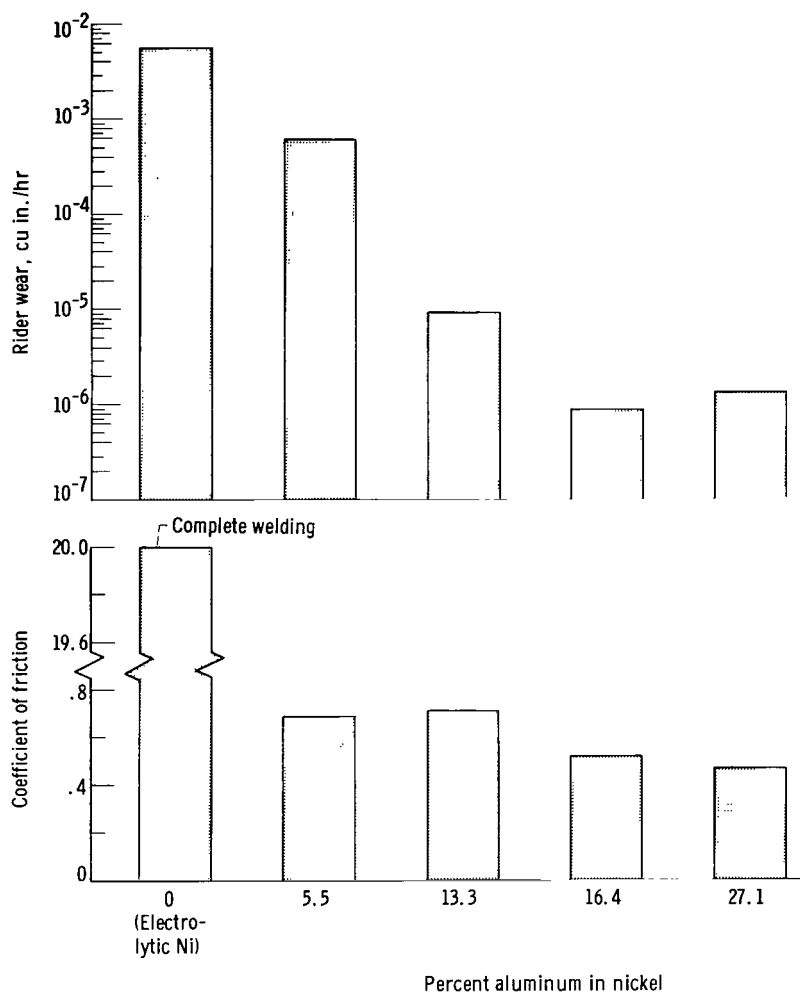
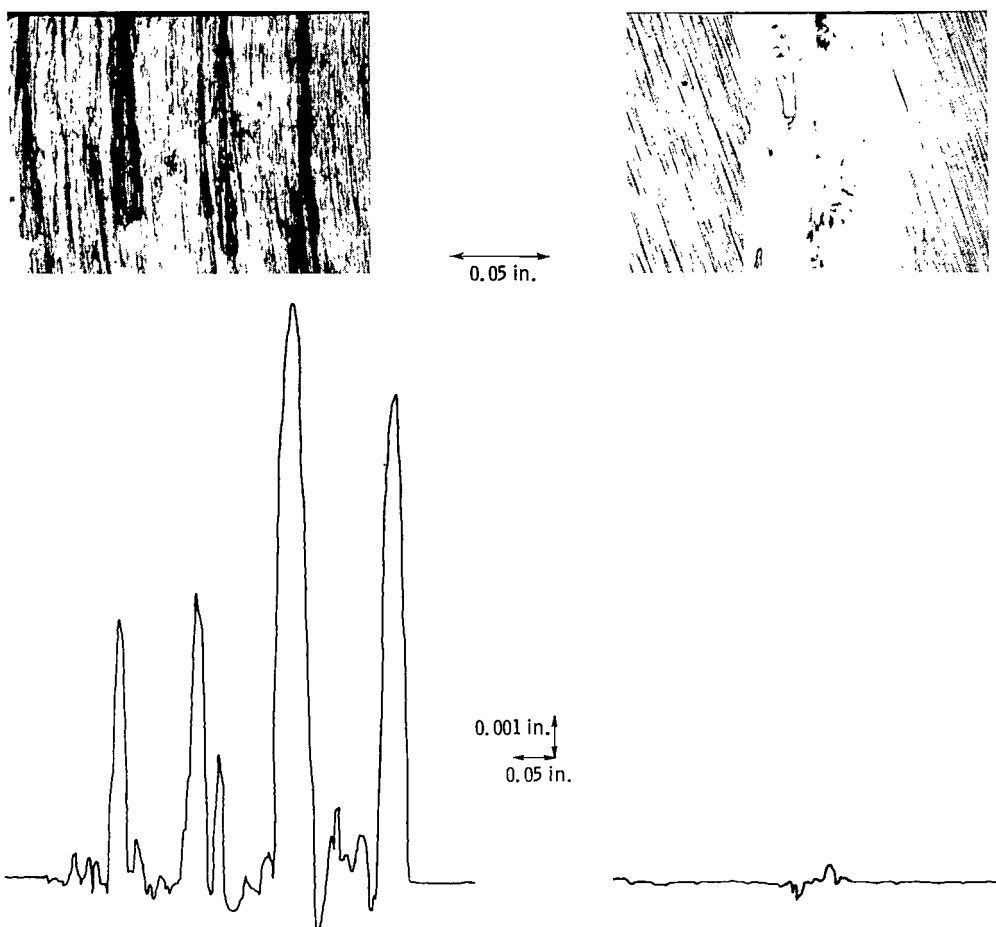


Figure 10. - Coefficient of friction and rider wear for various nickel-aluminum alloys in vacuum (10^{-5} mm Hg). Sliding velocity, 390 feet per minute; load, 1000 grams; ambient temperature, 75° F; duration, 1 hour; disk and rider of same material.

surface in vacuum decreases with increasing aluminum content for the two-phase structure. With the single-phase intermetallic NiAl, transfer and disk wear were observed. The results of figures 10 and 11 indicate that wear and metal transfer are less with the duplex intermetallic structure than with the simple intermetallic compound NiAl. This result is in accord with the mechanism discussed for such structures in reference 6.

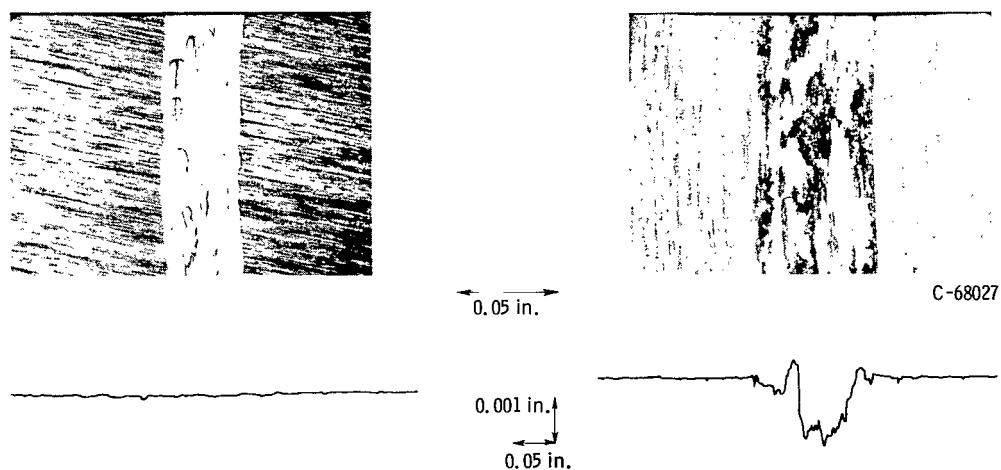
With a two-phase alloy system, where the mechanical properties of the two phases differ, the softer phase is smeared over the harder phase in the process of sliding much like lead is spread in a leaded bronze. The harder phase lends good mechanical properties to the structure, while the softer furnishes the lubricant or film that reduces metal transfer and wear.

In order to obtain comparative data on commercial nickel alloys, some friction and wear experiments were conducted in vacuum with two commercial



(a) 5.5 Percent aluminum in nickel.

(b) 13.3 Percent aluminum in nickel.



(c) 16.4 Percent aluminum in nickel.

(d) 27.1 Percent aluminum in nickel.

Figure 11. - Photomicrographs and surface profile traces of wear areas in disks of various nickel-aluminum alloys. Sliding velocity, 390 feet per minute; load, 1000 grams; ambient pressure, 10^{-9} millimeter of mercury; duration, 1 hour; disk and rider of same material.

nickel-base alloys. The composition of the nickel-chromium alloy was

Cr	Fe	C	Si	Al	Mn	Cu	Nb	Ti	Ni
15.0	7.0	0.4	0.4	0.7	0.5	0.2	1.0	2.5	Bal.

and that of the nickel-chromium-aluminum alloy was

Co	Cr	Fe	C	Si	Mo	Al	Mn	Ti	Ni
11.0	19.0	5.0	0.12	0.5	9.5	1.5	0.1	3.0	Bal.

The results obtained with these two alloys together with that for the 16.4-percent-aluminum - nickel alloy are presented in figure 12. In figure 12, the friction coefficient for the nickel-aluminum alloy (0.5) was approximately half that obtained for two commercial nickel alloys (1.0) in vacuum. The rider wear

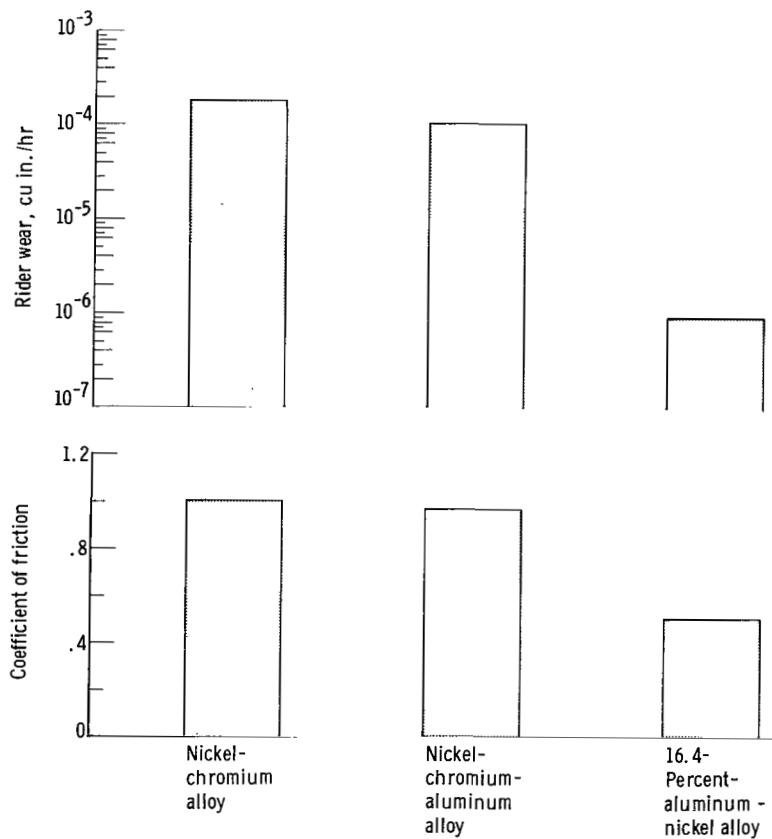


Figure 12. - Coefficient of friction and rider wear for various nickel-base alloys in vacuum (10^{-9} mm Hg). Sliding velocity, 390 feet per minute; load, 1000 grams; ambient temperature, 75° F; duration, 1 hour.

was two orders of magnitude less for the simple nickel-aluminum alloy than for the two commercial nickel alloys. Furthermore, metal transfer was obtained with the two nickel-base commercial alloys in vacuum, as indicated by the photomicrographs and the surface profile traces of figure 13. Essentially no

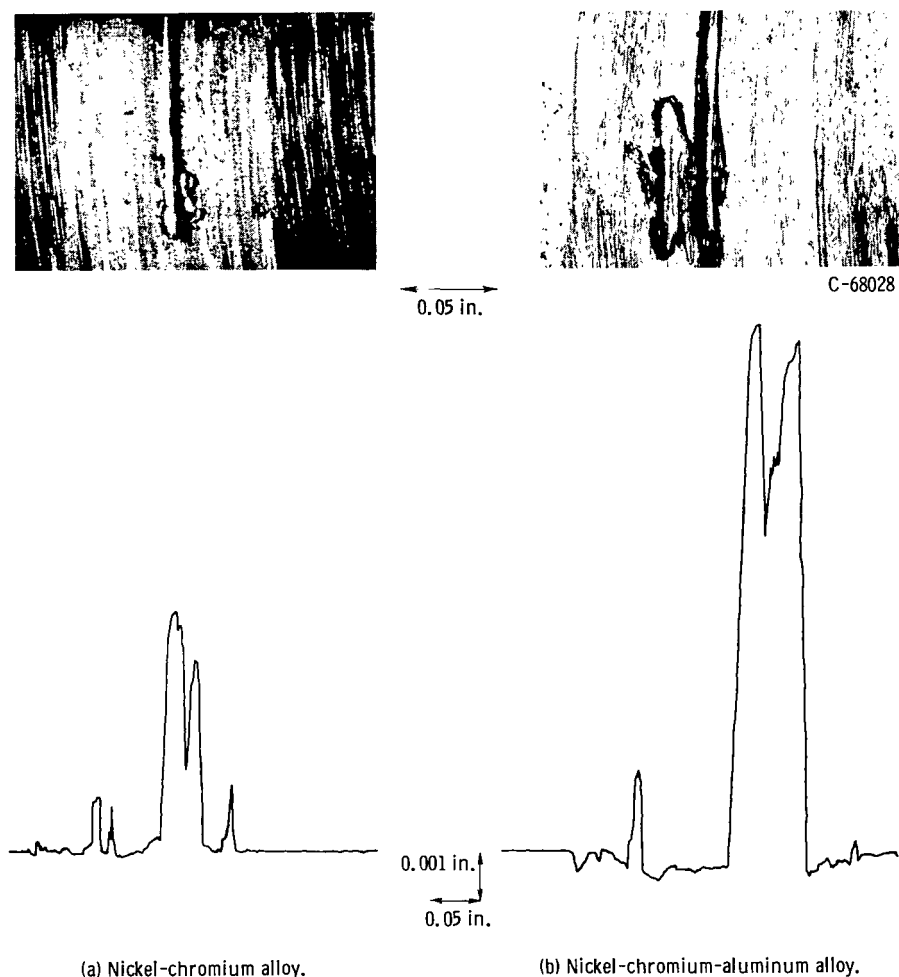


Figure 13. - Photomicrographs and surface profile traces of wear areas in nickel-chromium and nickel-chromium-aluminum disks. Sliding velocity, 390 feet per minute; load, 1000 grams; ambient pressure, 10^{-9} millimeter of mercury; duration, 1 hour; disk and rider of same material.

metal transfer was observed after vacuum experiments with the simple binary alloy (fig. 11).

Although friction, wear, and metal transfer characteristics for the simple binary nickel-aluminum alloys made them appear very attractive as slider materials, they are only binary alloys. Consequently, the question may be raised as to the other mechanical properties of these simple alloys. According to reference 10, the mechanical properties of the 17.5-percent-aluminum - nickel alloy are sufficiently good to indicate it has potential as a high-temperature alloy. The 16.4-percent-aluminum - nickel alloy had a hardness R_C of 36,

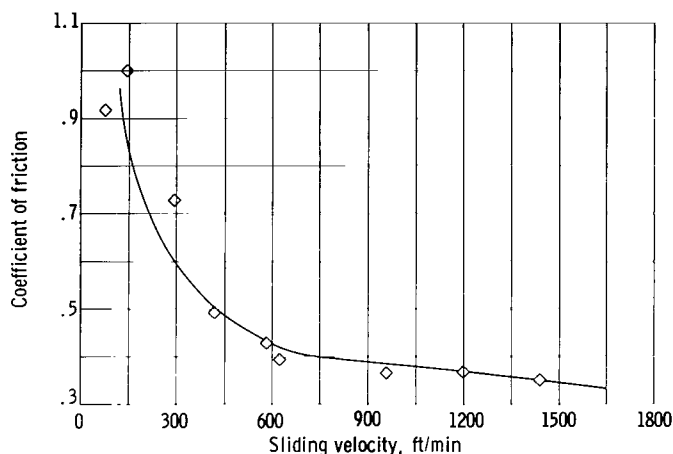


Figure 14. - Coefficient of friction for 16.4-percent-aluminum - nickel alloy in vacuum (10^{-9} mm Hg). Load, 1000 grams; ambient temperature, 75° F; disk and rider of same material.

while that of the nickel-chromium alloy was about 30 and that of the nickel-chromium aluminum alloy was 40.

In order to determine the effects of varied surface conditions on the behavior of the 16.4-percent-aluminum - nickel alloy, a friction experiment was conducted in vacuum over a range of sliding velocities. The results obtained in this experiment are presented in figure 14. Increasing the sliding velocity reduced markedly the friction coefficient. The friction coefficient was 1.0 at 150 feet per minute. This value decreased to 0.40 at 640 feet per

minute and 0.35 at 1440 feet per minute. These results suggest that increasing the sliding velocity allows for the increased flow of one phase of the duplex structure over the other, which affords greater surface protection.

INFLUENCE OF INCLUSIONS ON FATIGUE

Frequently, in discussions of the fatigue of materials in the literature, reference is made to the influence of inclusions on fatigue life (refs. 11 to 21). In summarizing what has been said in regard to fatigue, it is felt that insufficient quantitative data exist in the literature to make generalizations regarding (1) the behavior of specific inclusions in a clean metal and the role of inclusions on fatigue, (2) the influence of cast as opposed to wrought structures on fatigue, and (3) the relation between rotary bending fatigue and bearing fatigue. Certainly, the fatigue literature does not provide an adequate basis to indicate that cast alloys containing relatively soft microinclusions cannot be used in rolling-contact members. Rather, there is sufficient knowledge of fatigue in this area to suggest that, while the fatigue problem must be considered, it should not deter the development of bearing alloys with microinclusions to inhibit surface welding tendencies.

SUMMARY OF RESULTS

From the data obtained in an investigation of the friction and wear of nickel-aluminum alloys and sulfur-modified steels in vacuum, the following summary remarks can be made:

1. The addition of 0.4 to 0.5 percent sulfur to 52100, 440-C stainless steel, and M-2 tool steel appreciably reduced surface welding normally encountered with these alloys in vacuum (10^{-9} mm Hg). In particular, it reduced friction and wear markedly for 440-C and somewhat less for the other alloys.

2. Simple binary nickel-aluminum alloys were prepared. One had improved friction and wear when compared with two commercial nickel-base alloys. Of the nickel-aluminum alloys examined, 16.4 percent aluminum in nickel (duplex structure) exhibited the most promising behavior for vacuum applications.

Lewis Research Center

National Aeronautics and Space Administration

Cleveland, Ohio, February 25, 1964

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